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Jawaharlal Nehru

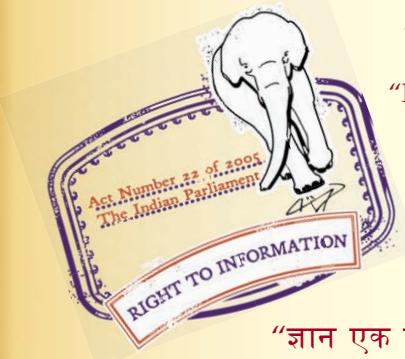
“Step Out From the Old to the New”

IS 7636 (1992): 4-Chloro-2-Toluidine [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

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“Knowledge is such a treasure which cannot be stolen”



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भारतीय मानक

4-क्लोरो-2-टोल्यूडीन — विशिष्ट

(दूसरा पुनरीक्षण)

Indian Standard

4-CHLORO-2-TOLUIDINE — SPECIFICATION

(*Second Revision*)

UDC 667.21 : 547.552 1'113

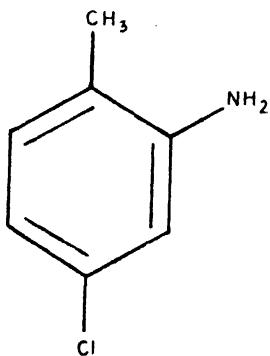
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FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

4-Chloro-2-Toluidine (C_7H_8NCl) is an intermediate used in the manufacture of dyes and pigments. Its hydrochloride is Fast Red KB Base which is used as a diazo component in azoic dyeing. It has the following structural formula:



4-CHLORO-2-TOLUIDINE

(Molecular Mass 141.5)

C. A. S. Registration No. [95-79-4]

This standard was issued in 1975. The Committee responsible for its preparation decided to revise it in order to update the requirements in accordance with the improved quality of the material being produced in the country. In the present version, the requirements of assay and crystallizing point have been revised and the requirement of impurities incorporated.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (revised)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

**AMENDMENT NO. 2 JUNE 2008
TO
IS 7636 : 1992 4-CHLORO-2-TOLUIDINE —
SPECIFICATION**

(First Revision)

(Page 1, clause 2) — Substitute 'IS 2552' for 'IS 3552'.

(PCD 9)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 1 MAY 1993
TO
IS 7636 : 1992 4-CHLORO-2-TOLUIDINE—
SPECIFICATION
(Second Revision)

[*Cover page, Title (Both in English and Hindi), Foreword, line 1, page 1, Title*] — Substitute 'First Revision' for 'Second Revision'.

(PCD 11)

Reprography Unit, BIS, New Delhi, India

*Indian Standard***4-CHLORO-2-TOLUIDINE — SPECIFICATION**
*(Second Revision)***1 SCOPE**

This standard prescribes the requirements and the methods of sampling and test for 4-chloro-2-toluidine.

2 REFERENCES

The following Indian standards are necessary adjuncts to this standard:

IS No.	Title
1070 : 1992	Reagent grade water — Specification (third revision)
1997 : 1982	Burettes (second revision)
3552 : 1989	Steel drums (galvanized and ungalvanized) (third revision)
5299 : 1969	Methods of sampling and tests for dye intermediates

3 REQUIREMENTS**3.1 Description**

The material shall be in the form of pale yellow coloured liquid. It becomes reddish brown on storage.

3.2 The material shall also comply with the requirements given in Table 1.

Table 1 Requirements for 4-Chloro-2-Toluidine

Sl No.	Characteristics	Requirements	Method of Test
(1)	(2)	(3)	(4)
i)	Crystallizing point, °C, Min	22.0	7.1.2 of IS 5299 : 1969
ii)	Assay, percent by mass, Min	98.5	Annex A
iii)	5-chloro-2-toluidine content, percent by mass, Max	0.2	Annex B
iv)	3-chloro-2-toluidine content, percent by mass, Max	0.2	Annex B

4 PACKING AND MARKING**4.1 Packing**

The material shall be packed in steel drums (see IS 2552 : 1989) or as agreed to between the purchaser and the supplier.

4.2 Marking

Each container shall bear legibly and indelibly the following information:

- Name of the material,
- Indication of the source of manufacture,
- Gross and net mass,
- Batch number, and
- The minimum cautionary notice worded as under:

'DANGER ! HAZARDOUS LIQUID AND VAPOURS RAPIDLY ABSORBED THROUGH SKIN'

4.2.1 The containers may also be marked with the Standard Mark.

5 SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed in 3 of IS 5299 : 1969.

5.2 Number of Tests

5.2.1 Tests for crystallizing point and assay shall be conducted on each of the individual samples.

5.2.2 Tests for the determination of remaining characteristics, namely 5-chloro-2-toluidine content and 3-chloro-2-toluidine content shall be conducted on the composite sample.

5.3 Criteria for Conformity**5.3.1 For Individual Samples**

The lot shall be declared as conforming to the requirement of crystallizing point and assay if each of the individual test results satisfies the relevant requirement given in Table 1.

5.3.2 For Composite Sample

For declaring the conformity of the lot to the requirements of description and all other characteristics (see 5.2.2), tested on the composite sample, the test results for each of the

characteristics shall satisfy the relevant requirements given in 3.1 and Table 1.

6 TEST METHODS

6.1 Tests shall be carried out according to the methods prescribed in col 4 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, 'pure chemicals' and distilled water (see IS 1070 : 1992) shall be employed in tests.

NOTE -- 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[*Table 1, Item (ii)]*

METHODS OF DETERMINATION OF ASSAY

A-1 OUTLINE OF THE METHOD

A known quantity of the material is titrated with sodium nitrite solution in acid medium. The amount of sodium nitrite solution consumed is expressed in terms of the amount of the amine present.

A-2 REAGENTS

A-2.1 Sodium Nitrite Solution — 0.1 N. This solution shall be freshly standardized against pure sulphanilic acid.

A-2.2 Concentrated Hydrochloric Acid — 30 percent (*m/v*).

A-2.3 Starch Iodide Test Papers

A-2.4 Potassium Bromide Solution — 25 percent (*m/v*).

A-3 APPARATUS

A-3.1 Mechanical Stirrer

A-3.2 Burette — *see* IS 1997 : 1982.

A-3.3 Beakers

A-3.4 Thermometer — Suitable to measure temperature in the range of 0° to 5°C.

A-4 PROCEDURE

A-4.1 Weigh accurately about 1 g of the sample and transfer into a 1 000 ml beaker. Dissolve in 500 ml of water and 50 ml of concentrated hydrochloric acid and heat if necessary. Add crushed ice into the beaker and stir the mixture mechanically until the temperature is about 0° to 5°C. While stirring mechanically, add standard sodium nitrite solution from the burette as rapidly as the spot test permits, keeping the temperature below 5°C. Test the solution by spotting on starch iodide paper. If the rate of diazotization is slow, add a few ml of potassium bromide solution to obtain sharper end point. The end point is reached if an immediate faint blue coloured ring appears when the solution is spotted on starch iodide paper and which persists for a period of 5 minutes or so without further addition of nitrite solution.

A-5 CALCULATION

$$\text{Assay, percent by mass} = \frac{V \times N \times 14.15}{M}$$

where

V = volume, in ml, of sodium nitrite solution used;

N = normality of sodium nitrite solution; and

M = mass, in g, of the sample taken for test.

ANNEX B

[*Table 1, Items (iii) and (iv)]*

DETERMINATION OF 3-CHLORO-2-TOLUIDINE AND 5-CHLORO-2-TOLUIDINE

B-1 OUTLINE OF THE METHOD

3-Chloro-2-toluidine and 5-chloro-2-toluidine are determined by thin layer chromatography.

B-2 REAGENTS

**B-2.1 3-Chloro-2-Toluidine- } prepare 0.002
pure reference } percent**

**B-2.2 5-Chloro-2-Toluidine- } 0.004 percent,
pure reference } 0.008 percent
solutions in
methanol**

B-2.3 Eluent — benzene : hexane (80 : 20)

B-2.4 Nitrous Fumes Chamber

A glass chamber of size 250 mm \times 250 mm \times 250 mm containing approximately 50 ml of concentrated HCl (30 percent *m/v*). The nitrous fumes are generated by adding a little solid sodium nitrite everytime before inserting the plate.

B-2.5 Spray Reagent — 0.1 percent (*m/v*) solution of American Base (N-1-Naphthylethylene-diaminedihydrochloride) in methanol.

B-3 APPARATUS

B-3.1 Thin Layer Chromatographic Chamber — 250 mm \times 100 mm \times 250 mm.

B-3.2 Micropipette of 10 Microlitre Capacity

B-3.3 Pre-Coated Thin Layer Chromatography Plates

TLC plates (200 mm \times 200 mm) coated with silica gel, about 0.25 mm thickness and with a fluorescence of F-254 nm.

B-4 PROCEDURE

B-4.1 Weigh accurately about 2 g of the sample (100 percent basis) and dissolve in 100 ml methanol. Take a plate coated with silica gel and spot 10 microlitre of each of the test solution and reference solutions along the start line at a distance of 3 cm from one another. Saturate the chromatographic chamber with eluents. Insert the plate in the chamber and allow to run for about 25-30 minutes during which time the solvent front travels approximately 10 cm past the start line. Remove the plate from the chamber and dry it in air. After drying put the plate in the nitrous fumes chamber for about 30 seconds. Remove the excess nitrous fumes by blowing air on it. Spray the plate with American base (A-2.5) when the spots characteristic of 3-chloro-2-toluidine and 5-chloro-2-toluidine become visible at *Rf* 0.73 and 0.33 respectively.

B-4.2 Reporting

Estimate the quantities of the impurities by comparing the colour intensity of the spots of the material under test with those on the reference samples.

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